RADIATE
Ion Beam Analysis
Quality Assurance Manual

ISSUE STATUS

Issue	Amendment/Comment	Author	Approved
1	Creation of amalgamated IBA QA documents from partners	RPW (Dec 2019)	
1.1	Corrected typos and generally improved text	CC (Dec 2019)	

Contents

1.   Introduction

This document covers the quality assurance procedures at the ion beam analysis facilities providing Transnational Access as part of the RADIATE consortium

RADIATE is an INFRAIA (Integrating and opening research infrastructures of European interest) project in H2020 with the main objective to provide user access to Europe's key ion beam facilities. More than 15.600 hours in total of trans-national access are offered to users at 11 ion beam facilities in Europe during the project duration of 4 years. The portfolio of the ion beam facilities ranges from specialized low-energy ion beam systems to large accelerators far beyond laboratory scale. They are operated as user facilities offering access to very specialized analysis and materials modification techniques for users from academia and industry. The facilities offer ion beam services in two application areas: (i) ion beam analysis; and (ii) ion beam modification.

This manual specifically covers ion beam analysis. A separate manual covers ion implantation and modification. Bespoke implantation experiments and analysis may also require their own separate Quality Assurance (QA) procedures on an "as required" basis.

RADIATE QA meetings take place twice per year via video conference. The results of QA testing must be reported at these meetings and any proposed adjustments to the QA procedures should be discussed there prior to implementation.

The following text outlines the QA procedures and their justification for Ion Beam Analysis.

2.   Ion Beam Analysis QA

Ion Beam Analysis (IBA) is an enabling technology for thin film scientists and engineers. It is a powerful group of analytical techniques for determining the elemental and isotope composition of thin films and other prepared materials. It can generate accurate (and traceable) analyses, with good spatial resolution both laterally and in depth.

IBA techniques have recently become much more powerful, since the self-consistent analysis of photon emission (PIXE) spectra together with particle scattering (RBS, EBS, ERD, NRA) spectra has become available.

The procedures in this section are based on procedures adopted and used by the Transnational Access providers in RADIATE for Ion Beam Analysis.

Techniques covered are:

- STIM: scanning transmission ion microscopy
- IBIL: ion beam induced luminescence
- PIXE: particle-induced X-ray emission
- RBS: Rutherford backscattering spectrometry
- EBS: elastic (non-Rutherford) backscattering
- ERD: elastic recoil detection
- NRA: nuclear reaction analysis
- PIGE: particle-induced gamma ray emission
- IBIC: ion beam induced charge
- RBS-c: RBS with channelling
- PIXE-c: PIXE with channelling
- MeV-SIMS: Secondary Ion Mass Spectrometry
- AMS: Accelerator Mass Spectrometry

2.1 General Requirements

During each run (a measurement period in the same beamline, usually 1 – 2 shifts of 8 hours), all experiment relevant parameters such as the accelerator and beam line settings, the detector calibration parameters and beam spot size must be recorded. Currently, there is no RADIATE database definition and the data are collected on an ad-hoc basis, often on paper logbooks for each beamline and accelerator.

The data must be recorded after each run and the system must be returned to the standard set up for the next user. The update of the database has to be done by the beamline scientist. Each beamline should have a checklist for starting, running and finishing the experiments. In addition, logfiles should be created automatically by the measurement programs to be able to check the conditions/parameters for the data-files. Data should be stored and retained in line with the RADIATE data management plan.

2.2 Ion Beam Analysis QA schedule

Each accelerator normally has more than one beam line dedicated to different Ion Beam Analysis techniques or sample types. Each beamline will normally have its own detector(s) that are in general not interchanged. Data on beamline and detector performance might also obtained and collected in different ways, specific for each application.

For this document to apply to the many diverse situations available in each of the laboratories the systems are separated into the different components: Accelerator (section 3); Beamlines (section 4); Detectors (section 5); Calibration materials (section 6) and Methods (section 7).

Each Quarter, relevant data summarised from the calibration data above, such as detector resolution, beam energy calibration factor, etc. is viewed to observe potential degradation of detectors and unexplained changes in the accelerator, which might affect future performance.

The following have been considered:

1) The Accelerator
   a. The absolute beam energy
   b. Energy stability
   c. Beam current stability
2) The beam line components (where they exist)
3) Detectors
   a. Solid State Particle detectors
   b. Gas particle detectors
   c. X-ray detectors
   d. Gamma ray detectors
4) Technique Specific data

3. <u>Accelerator</u>

3.1 Absolute Energy

Knowledge of the absolute energy is essential for depth profiling and quantification using particle scattering. It is also important for PIXE since the ionisation cross-sections are also

sensitive to the absolute energy. The accelerator energy is controlled by the generating voltmeter (GVM). The GVM is known to have an uncorrected temperature coefficient and therefore the laboratory should be maintained at a constant, monitored, temperature of 21 ± 2 °C.

3.2 The Protocol for Measuring Absolute Energy

A procedure for precisely determining the absolute energy using a resonant reaction has been specified in detail by Colaux, Terwagne and Jeynes (2015)[1]

Appropriate energy calibration points must be selected based upon the energy range of the accelerator and the experiments being performed. Instructions on how to carry out this calibration are given in detail in this paper. This procedure should be adopted and carried out so that the absolute energy can be precisely determined, together with the laboratory temperature, quarterly and in any case, whenever the GVM mechanism is disturbed.

The following resonant reactions can be used as to calibrate the absolute energy, other suitably sharp, resonances can also be used:

3.2.1   6 MeV

The absolute energy can be determined from the $H(^{15}N,\gamma\alpha)^{12}C$ reaction with a very sharp resonance at 6.385 MeV, often used for measurements of the hydrogen concentration. Where appropriate this resonance can be used to determine the absolute energy by performing an energy scan on a Si sample with some monolayers of H-containing water/organic contaminants on the surface.

3.2.2   3 MeV

A procedure for precisely determining the absolute energy using the resonance at 3038 keV of the $^{16}O(\alpha,\alpha)^{16}O$ reaction has been specified by Colaux[1].

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[1] J L Colaux, G Terwagne, C Jeynes, On the Voltage Calibration of Electrostatic Accelerators, *Nuclear Instruments & Methods B* **349** (2015) 173-183.

3.2.3   2.5 MeV

Non-Rutherford resonances – for example, in 2.5 MeV proton spectra from a lead-glass BCR126A certified standard, the $^{28}Si(p,p)^{28}Si$ resonance at 2088 keV is usually clearly visible, which approximately determines the beam energy.

3.2.4   1.8 MeV

The reaction $^{7}Li(p,n)^{8}Be$ with a threshold at 1880.6 keV can also been used[1].

3.2.5   1 MeV

Another, well-established and accepted reactions $^{27}Al(p,\gamma)^{28}Si$ at 991.8 keV (width 0.1 keV) is described in the paper.

3.2.6   430 keV

The $^{15}N(H, \gamma\alpha)^{12}C$ reaction with a sharp resonance at 429 keV can be used in the same way.

The energy should be confirmed (using a calibration standard such as Pb glass or by any other method) whenever convenient and logged, together with the laboratory temperature.

These data are collated on a quarterly basis and analysed for temporal variations.

3.3 Energy Stability

The short-term energy stability ("ripple") of the beam is an essential parameter in micro/nanobeam applications since the focussing lenses have very strong chromatic aberration. Long-term energy stability ("drift") is a problem since beam current is lost if the beam drifts across the apertures. Fluctuations in beam energy have been observed as a result of the GVM bearings malfunctioning. In these circumstances the GVM must be repaired.

3.3.1   Protocol for Monitoring Beam Energy Stability

The beam energy is a direct product of the terminal voltage. The terminal voltage, where possible, should be logged automatically by the accelerator control system. Where automatic

data logging is not possible values should be recorded manually once or twice each day. Fluctuations in the terminal voltage should be monitored and reported to the QA meeting.

The beam energy deduced from nuclear methods (above) is to be logged after each run.

These data are collated on a 3-monthly basis and analysed for temporal variations.

3.4 Beam Current Stability

There are several reasons why the current can show a long-term drift, like drift of power supplies, drift of the beam energy or changes in the ion source. A robust and continuous measurement of the current is therefore useful for highlighting potential issues.

3.4.1 Beam Current Measurement

Measurement of beam current accurately can be challenging as target materials can be insulating which will prevent charge monitoring directly through the sample holder or can emit secondary electrons which, if not taken account of, will create an error in the current measurement.

Target Holder: If the ion current is measured on the target holder then secondary electron suppression in front of the sample is essential. For insulating samples that charge-up, this will still be incorrect and alternative mechanisms are recommended.

Beam Chopper: A gold plated rotating vane which intercepts part of the beam and the backscattered ions are detected with a solid-state detector. The rate of the backscattered ions can be used to provide a relative measure of the beam current, which can be directly related back to a beam current via calibration by direct measurement into a Faraday cup.

Faraday Cup: A drop in or off-axis Faraday cup can be utilised to monitor beam current on a regularly (every few seconds) basis.

The actual method used will depend upon a number of constrains associated with different experiments and beam line conditions. However, in all cases, the average beam current will be monitored and record for each experiment.

4. Beam Line Components

Beam line performance is an important aspect of QA since it is important to ensure that the equipment is being used in the most efficient way and to identify any faults. Data on beam line performance (from the logged data) will be collated and reported to the QA meeting every three months. The performance data collected per beam line will depend upon the line and its application. The minimum data monitored for each line will be the beam current.

Additional performance date will be expected for focussed or collimated microbeams. The beam spot size should be measured using either the Cu Kα X-ray line from a Cu SEM calibration grid, or by use of specially lithography prepared calibration samples. This data should be logged along with the slit and quadrupole settings.

5. Detectors

The laboratories possess multiple particle, X-ray (Si(Li) or SDD) and gamma-ray (HPGe or SDD) detectors. The performance of the detectors is integral to obtaining accurate analysis of samples and to pre-empt the need for detector replacement. Detector calibration parameters that should be recorded are as follows.

5.1 Particle Solid State Detectors

The electronic gain and detector dead layer thickness should be determined using a suitable calibration standard (usually thin Au/Ni on SiO_2/Si or BCR 126A Pb glass) for each particle detector and recorded for each run. The calibration spectra reference should also be logged. The FWHM of the detector, together with the FWHM of the electronic pulser (to monitor the noise behaviour of the detection channel) should also be recorded in the database for each run. The protocol for this calibration is described at length in the literature[2,3].

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[2] J L Colaux, C Jeynes, High Accuracy Traceable Rutherford Backscattering Spectrometry of Ion Implanted Samples, *Analytical Methods* **6** (2014) 120-129.
[3] J L Colaux, C Jeynes, Accurate Electronics Calibration for Particle Backscattering Spectrometry, *Analytical Methods* **7** (2015) 3096-3104

5.2 Particle Gas Detectors

The FWHM of the detector, together with the FWHM of the electronic pulser (to monitor the noise behaviour of the detection channel) should be recorded in the database for each run.

5.3 X-Ray Detectors

The effective filter thickness and the effective distance, together with the standard deviation of the measured/certified compositions of X-rays coming from the Pb glass calibration standard (excluding Fe Kα, which has a high uncertainty) should be recorded for each run.

5.4 Gamma Detectors

A background spectrum should be recorded in the database for each run, and each of the major peaks identified and used for calibration.

6.   Calibration samples

In this document, specific samples are named that are currently used for the calibration. But they may be replaced by other samples if they become too old or damaged or better ones become available.

Often used standards for Ion Beam Analysis:

Techniques	Material	Notes
PIXE	NIST 1412, 612, 620, high purity metals	Thick NIST are glass standards
	NIST 2783, MicroMatter XRF standards	thin; NIST is the aerosol reference sample
	BCR126 (lead glass)	Used in PIXE, PIGE & RBS
PIGE	NIST 1412, 620	thick, glass standards (Na, B, Li)
	^{132}Ba, ^{152}Eu, ^{222}Rn	calibration sources
RBS	IRMM-302	Sb implanted in Si/SiO_2
	High purity metals; MM XRF standards	Thick and thin

	SPIRIT Calibration sample	As implanted in Si/SiO$_2$
	AuNi/SiO$_2$/Si	As per reference 1
ERD	Kapton	
	BAM 100	a-Si:H reference sample for the hydrogen. Reference value for the hydrogen content is (12.2±0.5) at.%
	NIST 620	For heavier elements

7. Method specific

7.1 Accelerator Mass Spectrometry

Besides the QA requirements common to all ion beam methods, AMS is especially sensitive to good and well-tuned ion optics of the whole instrument. As the sample is placed in the ion source, beam losses can distort the primary measurand, the intensity ratio between beams. The AMS groups thus follow determined protocols on how the ion beam is tuned. The establishment and approval of such a protocol is an integral part of the development a new isotope capability.

At the end of the tuning process, and throughout the measurement, the performance is verified with known standard materials. This is achieved by measuring all samples, unknowns and standards, repeatedly and in alternation, until the required precision is achieved, or the sample is used up. The repeatability of the measurements is included in the estimation of the uncertainty.

Reference materials are used both to assess machine performance but may also go through the whole sample preparation and measurement process. Among these standard materials, so called "blanks" (i.e. samples without any content of the isotope of interest) play a special role; they allow to assess the instrument and chemical background, which is in many cases the limiting factor for measurement sensitivity. These standards are also used to normalize the AMS measurement and allow a blank correction

Following generally adopted practice of environmental analyses, together with every batch of samples, the users must provide quality control materials. These are typically international reference materials (NIST, IAEA, ...; e.g. sea water, soil, ...) which are as similar as possible

to the unknown samples and also go through the whole sample preparation. Their results are not used for the evaluation of the unknown samples but serve as independent quality control.

All steps of the measurements are documented, either manually in the instrument logbook, or electronically.

The following section explains the specific implementation at the different facilities.

7.1.1   UNIVIE

Check lists exist for all isotopes; the lists are online editable to allow easy addition of new findings and warnings concerning new problems or uncommon instrument configuration. The newest version is printed out for every measurement, and with check marks and notes archived after the measurement.

Ion optics fine tuning is done automatically. So-called "Flat Tops" are acquired for all machine parameters (i.e. dependency of ion optical transmission on parameter value). The data is archived and accessible through the web interface. For ^{14}C measurements, the data is already shown in comparison to a so called "golden setup" as a reference. The establishment of such a "golden setup" is also ongoing for the other isotopes. An automatically created, printable "Tuning report" with all diagnostic data and diagrams is planned.

The machine status is monitored by two specialized software components: a Cumulative Status Manager checks critical (and prone-to-fail) components several times per second, and pauses the measurement if an abnormal condition is detected. A so-called Diagnose Demon checks several hundred machine parameters regularly about every five minutes and warns the operator about suspicious values.

Parameter logging visualizing: critical parameters (e.g. vacuum, ion source and pelletron behaviour) are electronically logged; plots are online accessible through a web interface.

The following reference materials are used:

- ^{14}C: Reference Materials from NIST, IAEA; In-house standards Vienna-CTW2, Vienna-CTW0, lignite

- ^{236}U: Vienna-KkU, in-house standards Vienna-KkU-D30, Vienna-US8, Vienna-US8-D30

- ^{10}Be: SMD-Be-XX, SM-Phenakite (Silke Merchel, HZDR); S555N (ETHZ)

- ^{129}I: In-house standards Vienna-IS11, Vienna-AgI_111, Vienna-NaI_1

- ^{26}Al: In-house Standards M-XX from LMU/TU Munich; SM-Al-XX (Silke Merchel, HZDR); AW-V-XX, AW-M-XX (Anton Wallner, ANU)

- ^{36}Cl: SM11,12,13 (Silke Merchel, HZDR)

Additional to the reference materials used for quality control, we routinely remeasure archived samples from previous sample campaigns, and we encourage users to send a limited number of doublets from unknown samples, if enough material is available. We also appreciate "hidden" QA samples, i.e. doublets from unknown samples and additional reference materials, not identified before the measurement.

7.1.2   ETHZ

For every measurement campaign, we perform calibrations of the current integrators, check stripper and detector gas pressures etc.

Tuning is essentially assured when at the end of the process the performance is checked with known standard materials. This is logged in the logbook (and electronically).

During the measurement critical components (e.g. TV, cathode current) are recorded and the measurement is stopped if not ok. All machine parameters are logged during a measurement and a correlation analysis is done of scatter vs. ion-optical component. So that potential problems in the instrument can be identified.

For the ETH AMS standards and measurement procedures we usually refer to the paper by Christl *et al.*, 2013. Updated measurement procedures for some nuclides are taken from the newer papers Christl *et al.*, 2015 (^{236}U); Vockenhuber *et al.*, 2015a (^{41}Ca); Vockenhuber *et al.*, 2015b (^{129}I); Müller *et al.*, 2015 (^{26}Al).